

Magnetic-field-driven superconductor-insulator transition in granular In/InO_x films

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We have measured the differential resistance of granular In/InO_x films near the magnetic-field-driven superconductor-insulator (SI) transition. We observed the development of a sharp zero-bias peak in the differential resistance inside the superconducting gap in the field-driven insulating regime. We confirmed that the peak in the differential resistance originated from the Coulomb blockade of pair tunnelings between adjacent superconducting grains. Although almost full phase fluctuations are induced in a magnetic field which is far lower than the critical field of the SI transition, the global SI transition in granular systems is caused by additional phase fluctuations along with reduced pair tunnelings due to Coulomb blockade between superconducting grains. [S0163-1829(96)03830-1]

I. INTRODUCTION

It has been known that, *in zero field*, the amplitude of the superconducting order in uniformly disordered films is reduced with increasing disorder due to enhanced electron-electron interaction, leading to the second-order superconductor-insulator (SI) transition as the superconducting order vanishes.¹ In the case of granular systems, however, the local superconductivity always exists in the grains at sufficiently low temperatures and the global superconducting order is determined by the interplay between the intergranular Josephson coupling and the grain charging effect.² With increasing junction resistance R_J in a single Josephson junction between two grains in the systems (or equivalently, increasing system disorder), the Josephson coupling energy expressed at low temperatures ($k_B T \ll \Delta_c$) as $E_J = \hbar I_c / 2e = (1/2)(R_Q / R_J) \Delta_c$, where I_c is the junction critical current and Δ_c is the superconducting gap energy in the grains, can become smaller than the charging energy $E_c = e^2 / 2C_0$ for small enough grains, where C_0 is the grain capacitance. The resulting reduced fluctuations in intergrain charge transfer induce large fluctuations in the phase between the grains, which will hinder the formation of the global phase coherence. The competition between the intergranular Josephson coupling and the grain charging is well manifested in the quasireentrant behavior for films with the normal-state sheet resistance R_N close to the quantum resistance $R_Q (= h/4e^2)$.

Recently, much interest has been focused on the critical effect of a magnetic field on the destruction of superconductivity in two-dimensional systems,¹ both in uniformly disordered films and granular films. The application of a magnetic field enables one to tune continuously through the zero-temperature SI transition in disordered superconducting films with the normal-state sheet resistance R_N close to R_Q . A scaling theory^{3,4} indicates that, in contrast to the case of the transition induced by disorder, the field-tuned SI transition in the zero-temperature limit in disordered films is induced by increasing fluctuations in the phase, rather than the amplitude, of the superconducting order parameter, which is in good accordance with the recent experimental observations in various systems.⁵⁻¹⁰ The theory which may well be ap-

plied to either uniformly disordered systems or granular systems assumes that the localization of the condensed Cooper pairs due to extremely high mobility of the field-induced vortices at zero temperature brings about the SI transition. Since the essential assumption of the theory is the existence of the well-defined local superconducting order granular systems composed of grains which are large enough to support the bulklike superconducting order at low temperatures seem to be more relevant to test the boson-only scaling theory.^{3,4} To date the field-tuned SI transition in granular systems has been less studied than that in uniformly disordered systems. Preexisting experimental results¹¹ indicate that, in contrast again to the case of the transition induced by disorder, a strong transverse external magnetic field induces a suppression of the magnitude of the superconducting order in granular films. Thus, one of the issues is whether the magnitude of the superconducting order remains finite at the field-tuned SI transition in granular systems so that the transition is caused by the intergranular phase fluctuations. In this paper we address the issue by closely examining the temperature and magnetic-field dependence of the differential resistance (DR) in granular films near the field-driven SI transition. We find that the SI transition in granular systems is much subtler than was thought to be before.

II. EXPERIMENTAL DETAILS

Granular In/InO_x films were prepared by adopting the method as used in Refs. 7, 12, and 13. A layer of thickness Δd consisting of pure indium grains which were electrically separated from one another was thermally deposited in a high vacuum (the base pressure of the growth chamber: 5×10^{-7} Torr) on a substrate held at room temperature. The surface of a film was then oxidized in the oxygen partial pressure of 1×10^{-4} Torr for 4 min typically. The same procedure was repeated several times to make the total thickness of the sample be $d = 80$ Å. The nominal thickness of each indium layer was $\Delta d \approx 10$ – 16 Å. The advantage of fabricating granular films in this way is the easiness of regulating the thickness of oxide barriers between grains by varying the oxygen partial pressure during oxidation and the oxidation time, while keeping the size of the grains almost constant.

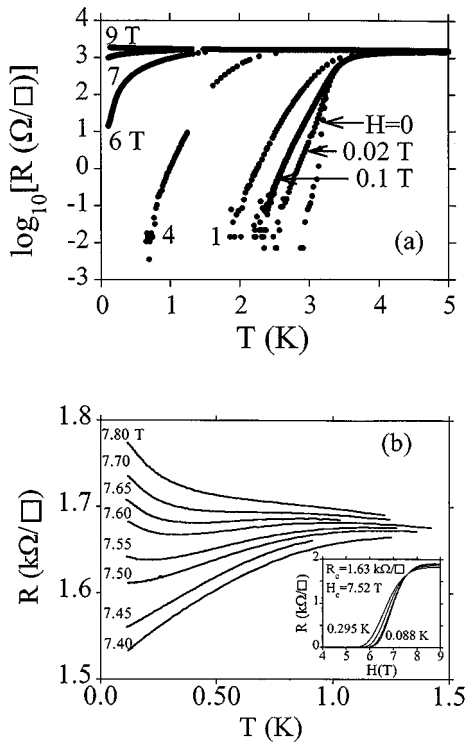


FIG. 1. (a) Temperature dependence of the sheet resistance in external magnetic fields ranging from 0 to 9 T. (b) Temperature dependence of the sheet resistance in external magnetic fields near the field-driven superconductor-insulator (SI) transition. Inset: the magnetic-field dependence of the resistance isotherms at 0.088, 0.130, 0.191, and 0.295 K near the SI transition, where the fixed point $(H_c, R_c) = (7.52 \text{ T}, 1.63 \text{ k}\Omega/\square)$ corresponds to a set of data with $dR/dT = 0$ in the low-temperature region in (b).

Microstructural analysis using scanning electron micrograph reveals that the lateral diameter of the grains is $a = 200\text{--}300 \text{ \AA}$. The average size of the grains prepared in this way turned out to be close to the one prepared by continuous thermal evaporation in the corresponding partial pressure of oxygen. In contrast to the case of continuous evaporation in the atmosphere of oxygen, however, one can regulate the size of the grains and the thickness of the oxide barriers separately using this method. The samples were patterned using a metal stencil into a Hall-bar shape for four-terminal measurements. The film was 0.4 mm wide and the distance between the voltage leads was 2.8 mm. The large-scale homogeneity of the film was checked by measuring the resistances over three separate sections along the length of the film, which agreed with one another within less than 3% of deviation. The sample resistance was measured, using both dc and ac phase-sensitive measurements at 33.5 Hz at temperatures ranging from 300 K to 100 mK and in a magnetic field up to 9 T. In order to increase the sensitivity of DR measurements a large background dc resistance was subtracted using a Kelvin-bridge circuit.

III. RESULTS AND DISCUSSION

Three samples were investigated in this experiment, among which the typical data for one sample are presented below. In Fig. 1(a) we illustrate the typical temperature de-

pendence of the sheet resistance (measured with $I = 100 \text{ nA}$) of the sample SI1 with varying magnetic fields applied perpendicular to the plane of the film. In zero magnetic field the R vs T curve exhibits a slight double-resistive transition near the tail, which is the hallmark of a granular superconducting film. The mean-field transition temperature, $T_c = 3.384 \text{ K}$, determined from the best fit to the Aslamasov-Larkin¹⁴ fluctuation conductivity, turns out to be very close to the value of pure bulk indium, 3.40 K. As the magnetic field increases the sample exhibits a number of characteristic features in the R vs T curve. In the low-field region ($H < 1 \text{ T}$) the superconducting transition broadens with increasing field, while the onset temperatures of the superconductivity are almost unaffected. In this region thermally activated resistances with field-dependent activation energy were observed. With increasing field in the range of $1 \text{ T} < H < 4\text{--}5 \text{ T}$, the mean-field transition temperature T_c is suppressed without much additional broadening of the transition width, which implies that the amplitude rather than the phase of the superconducting order in the grains is affected (suppressed) by the magnetic field. This looks rather counter-intuitive because, for this granular film in the high-magnetic-field range, the magnetic field apparently affects the amplitude of the superconducting order parameter more rather than its phase as observed in zero-field disorder-driven destruction of global superconductivity. This effect has been observed in other granular systems.¹¹ The crossover magnetic field separating the two regions corresponds to the field generating about one magnetic flux quantum $\Phi_0 = h/2e$ through the area $\sim \pi R^2$, where R is the mean radius of the grains. The mean radius of the grains estimated from the crossover field of $\sim 1 \text{ T}$ as observed in the sample is 250 \AA , which is very close to the value determined from the microstructure analysis, $100\text{--}150 \text{ \AA}$. Since the magnetic field of order of the crossover field gives almost maximum phase fluctuations any additional field beyond the value causes mainly the suppression of the superconducting order in the grains.

For a higher field between 7 T and 9 T, the film exhibits the field-tuned SI transition as shown in detail in Fig. 1(b). Small quasireentrance is seen in the fields between 7.5 T and 7.65 T, which is another manifestation of the granularity of the sample. The low-temperature (say 0.1 K) critical field, H_c , defined by the field at which the temperature gradient of resistance dR/dT vanishes, was 7.52 T. The corresponding critical resistance R_c was $1.63 \text{ k}\Omega/\square$, which was much smaller than the quantum resistance, $R_Q = 6.45 \text{ k}\Omega/\square$, as well as the critical value reported by other groups,^{5,6} presumably due to the difference in the morphology of the films. Recently it also has been argued⁸ that the existence of increased quasiparticles near the SI transition may cause the deviation of the critical resistance from the quantum resistance which was theoretically predicted to be universal. In our sample also the increased quasiparticle tunnelings by the field-induced suppression of the superconducting order near the SI transition may have caused R_c to deviate much from R_Q . At this point the question can be raised as to whether the amplitude suppression with increasing magnetic field will continue so that the order parameter eventually vanishes completely at the SI transition or it remains finite at the transition and the transition is driven by the phase fluctuations as predicted by the boson-only scaling theory.

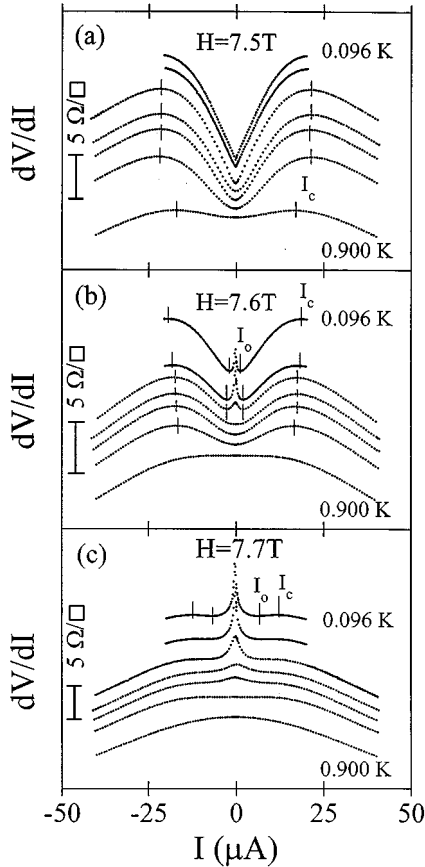


FIG. 2. Differential resistance per square of the sample as a function of the bias current at temperatures, 0.096, 0.136, 0.234, 0.403, 0.413, 0.596, and 0.900 K from top to bottom for external magnetic fields, (a) 7.5 T, (b) 7.6 T, and (c) 7.7 T. For clarity, each set of data is displaced vertically by an arbitrary amount. The vertical bars indicate I_c and/or I_0 as explained in the text.

The temperature independence of the resistance in a given magnetic field corresponds to a fixed point in the plot of the magnetic-field dependence of the resistance isotherms at 0.088, 0.130, 0.191, and 0.295 K as in the inset of Fig. 1(b). The fixed point for the sample SII is $H_c = 7.52\text{ T}$ and $R_c = 1.63\text{ k}\Omega/\square$, as mentioned above. Below, by closely examining the DR, we will focus on whether the superconducting order vanishes near the critical value H_c . The other noticeable feature in the inset is that the magnetoresistance in fields above the fixed point looks to increase very slowly even at the lowest temperature (0.088 K) and in the highest-field range ($\sim 9\text{ T}$) used in measurements. In fact, in the insulating regime in this high-field range a logarithmic temperature dependence of resistance was observed, which is a typical behavior of a weak localization (with e - e interactions) (Ref. 16) while the behavior is in contradiction to the variable-range-hopping behavior assumed by the scaling theory.³

Figure 2 shows the typical DR as a function of the bias current I for temperatures from 0.096 K to 0.900 K in a magnetic field of (a) $H=7.5\text{ T}$, (b) $H=7.6\text{ T}$, and (c) $H=7.7\text{ T}$, which are close to the range of the critical magnetic field determined from the field dependence of R vs T data in Fig. 1(b) and its inset. It also corresponds to the region of temperature and field where the quasireentrant behavior is most conspicuous in Fig. 1(b). The current modu-

lation (dI) for the measurements of DR was 50 nA. Each set of the data was shifted vertically by an arbitrary amount in the figure for the sake of clarity. Note that the bias current of 100 nA used to measure the R vs T data in Fig. 1 corresponds to almost zero bias in Fig. 2. In Fig. 2(b) distinct features develop with varying temperatures. At the highest temperature ($T=0.900\text{ K}$), the DR decreases monotonically with increasing bias current. In the temperature range below 0.596 K, however, the DR shows the Josephson-junction-like behavior (a increasing DR with bias) in the low-bias region, but crosses over to a decreasing DR with bias beyond a critical value I_c denoted by the vertical bars in the figure. The variation of the DR in Fig. 2, however, is almost 3 orders smaller than the background dc resistance as shown in Fig. 1(b), which implies the existence of large quasiparticle tunnelings in addition to pair tunnelings in the ranges of the temperatures and the magnetic fields. The magnitude of I_c decreases only slightly with increasing temperature below $T=0.596\text{ K}$, while it decreases rapidly between 0.596 K and 0.900 K. The variation of the increasing DR with the bias current becomes steeper with decreasing temperatures. But the variation of the decreasing DR with bias current sufficiently beyond I_c is very insensitive to the temperature change. Inside the region bounded by $\pm I_c$ a zero-bias peak develops at temperatures below 0.234 K and grows sharply with decreasing temperature. We suppose that the zero-bias peak arises from the Coulomb blockade of Cooper pairs and quasiparticles, since it arises inside the superconducting gap at a finite temperature while the former effect must be more dominant as the temperature is lowered. Even at low temperatures quasiparticle tunneling is expected to be fairly high because of amplitude fluctuations induced by a high external magnetic field.^{11,15} We assume that the onset of the Coulomb blockade corresponds to the minimum DR at I_0 , which is denoted by another set of vertical bars. In a field of 7.5 T as in Fig. 2(a) the DR in the same temperature range 0.096–0.900 K shows similar features as in Fig. 2(b), except for the fact in this case that no zero-bias peak appears and the variation of the increasing DR with field is steeper than in $H=7.6\text{ T}$. In contrast, in $H=7.7\text{ T}$ as in Fig. 2(c) the critical current (along with the superconducting gap) is much suppressed to about $I_c=12\ \mu\text{A}$ even at the lowest temperature 0.096 K and the DR becomes all decreasing at temperatures above 0.234 K, while the zero-bias Coulomb peak appears at temperatures as high as $T=0.413\text{ K}$. The Coulomb peak disappears with increasing temperatures due to the increased thermal activation of charge transfer over the Coulomb barrier.

In Fig. 3 the DR measured with a current modulation of 50 nA at $T=0.096\text{ K}$ is plotted as a function of bias current I for various magnetic fields between 7.5 T and 7.7 T in steps of 0.025 T. In the figure again we denote the critical bias currents of pair tunneling, I_c , and the Coulomb blockade, I_0 , as vertical bars. The values of I_c are shown to be suppressed rapidly with increasing field in such a narrow range near the critical field H_c . Here, we assume that the superconducting critical bias current can be converted into the superconducting gap voltage using the Ambegaokar-Baratoff¹⁷ relation $\Delta_c/e = (2/\pi)I_c R_N(T, H)$ at sufficiently low temperatures satisfying $k_B T \ll \Delta_c$. We also assume that the Coulomb gap at a given temperature and a

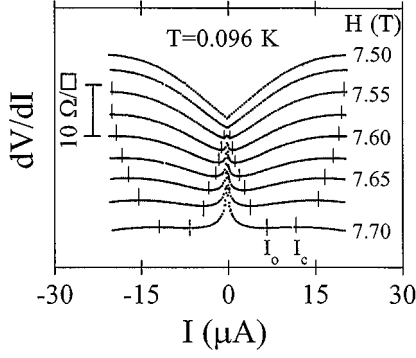


FIG. 3. Differential resistance per square of the sample as a function of the bias current at $T=0.096$ K for external magnetic fields ranging from 7.50 to 7.70 T by a step of 0.025 T from top to bottom. For clarity, each set of data is displaced vertically by an arbitrary amount. The vertical bars indicate I_c and/or I_0 as explained in the text.

field can be estimated from the relation $\Delta_0 = eV_0 = I_0 R_N(T, H)$, since the IV characteristics are almost ohmic as can be evidenced by the small variation of the DR as shown in Fig. 2 compared with the dc resistance in Fig. 1(b). The insulating Coulomb gap starts developing around $H_c = 7.55$ T and grows rapidly with increasing field. The value of this critical field for the onset of the insulating Coulomb gap at this temperature is very close to the one determined from the R vs T data.

We believe that the decreasing DR for the bias beyond I_c is caused by quasiparticle tunnelings above the superconducting as well as the charging gap between superconducting grains. In this case, the bias current makes the charging barrier tilted so that the tunneling conductance increases with increasing bias current (or voltage). The slope of the variation of the decreasing DR with bias in a given magnetic field in Fig. 2 is rather insensitive to the temperature, even below the critical temperature for the appearance of the superconducting gap since the tunneling of quasiparticles between superconducting grains above I_c follows the tunneling characteristics of normal electrons between normal grains. On the other hand, the onset of the insulating zero-bias peak is closely related with the sharp reduction of the charge transfer due to Coulomb blockade between superconducting grains, where the superconducting order in the grains remains finite. Thus in our granular films the transition to the insulating state in zero bias takes place while the local superconducting order persists in grains as assumed by the scaling theory. This is in agreement with the recent observation in granular Al films where a significant suppression of the local superconducting order was observed in a magnetic field of several tesla.¹⁸

We plotted V_0 and V_c at $T=0.096$ K, almost the lowest temperature used in the experiment, as a function of magnetic field in Fig. 4(a), where V_0 and V_c are obtained from the relations $V_0 = I_0 R_N(T, H)$ and $V_c = (2/\pi) I_c R_N(T, H)$. With increasing magnetic fields V_c decreases with a concurrent increase of V_0 . It is in accord with the assumption that V_c is the superconducting gap voltage and V_0 is the critical bias voltage for the onset of the Coulomb blockade. $V_c(H)$ shows the magnetic-field-dependent suppression of the su-

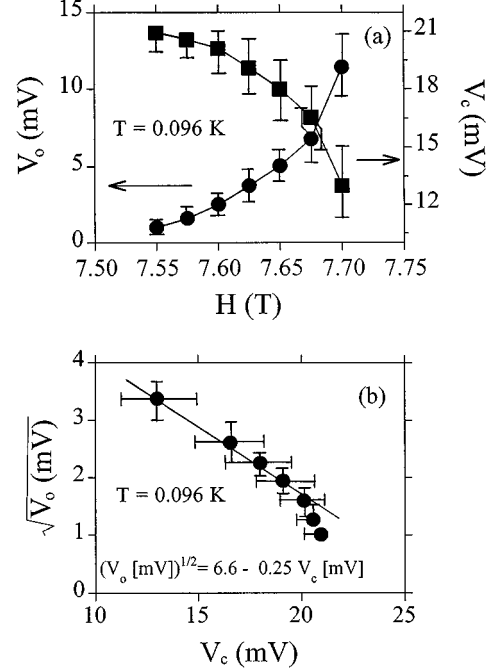


FIG. 4. (a) Magnetic-field dependences of the critical voltages per a square of the sample; V_0 (the onset voltage of Coulomb blockade near zero bias) and V_c ($=N\Delta_c/e$, where N is the number of grains connected in series along the length of a square of the sample). V_0 and V_c were obtained from the relation $V_0 = I_0 R_N(T, H)$ and $V_c = (2/\pi) [I_c R_N(T, H)]$, respectively. The lines connecting the data points are guides to the eye. (b) Functional dependence of V_0 on V_c , where the best fit satisfies the relation $[V_0 \text{ (mV)}]^{1/2} = 6.6 - 0.25 V_c \text{ (mV)}$.

perconducting gap. We can estimate the value of the blockade voltage V_0 in terms of a value of V_c by considering the energetics of Cooper-pair tunneling with Coulomb blockade in the following way. Here, for simplicity, we only consider pair tunnelings between superconducting islands, although we assume that quasiparticle tunnelings are non-negligible as mentioned above. When a pair tunnels from a neutral superconducting grain to an adjacent superconducting grain a bound pair of charge soliton is formed. We consider the energetics of an unscreened charge soliton pair in the limit of $T=0$ in a bias voltage V as

$$U(r) = 2E_0 - E_J - 2E_0 \frac{a}{r} - 2e^*(V/L)r, \quad (1)$$

where r is the separation between the charge-anticharge pair members, V/L is the average applied electric field along a sample, a is the average diameter of superconducting grains, and $e^* = 2e$. The first term with $E_0 = (e^*)^2 / (2C_0)$ is the grain charging energy to create a pair of nearest-neighbor Cooper-pair solitons and $E_J \approx (R_Q / 2R_N) \Delta_c = (R_Q / 2R_N) e^* V_c^{\text{single}}$ is the Josephson coupling energy of a junction formed between two adjacent superconducting grains, where C_0 is the self-capacitance of a grain and V_c^{single} is the superconducting gap voltage per junction which can be alternatively expressed as the total voltage along the length of a square of a sample (with length of $L = 0.4$ mm) divided by the number of grains N (≈ 16000) in it as

V_c/N . The third and fourth terms represent the attractive interaction between charge solitons of opposite polarities at a distance r due to the self-Coulomb field and external electric field, respectively. In Eq. (1) we neglected the thermal energy because at $T = 0.096$ K the thermal energy is at best about 10% of the grain charging energy. The potential energy in Eq. (1) has a saddle point at $r_c = (2E_0 a L / e^* V)^{1/2}$, with an energy barrier at the saddle of $U(r_c) = 2E_0 - E_J - 2(2E_0 e^* V a / L)^{1/2}$. In the zero-temperature limit a finite conduction due to pair tunneling can take place when the energy barrier is overcome by an external electric field, i.e., $U(r_c) < 0$. Thus, we get the relation between the onset voltage of conduction, V_0 , and the superconducting gap voltage V_c as

$$\sqrt{V_0} = \alpha - \beta V_c, \quad (2)$$

where $\alpha = (E_0 L / 2e^* a)^{1/2}$ and $\beta = (R_Q / 4R_N)(e^* L / 2E_0 a)^{1/2} / N$. In Fig. 4(b), $\sqrt{V_0}$ is plotted as a function of V_c , which shows a linear relationship in this plot as predicted by Eq. (2). We believe that the deviation of the data in the range of small V_0 from the above relation is due to a finite-temperature effect, which would have been more prominent for smaller values of V_0 . The values of α and β estimated from the data in the figure turn out to be $6.6 \text{ (mV)}^{1/2}$ and $0.25 \text{ (mV)}^{-1/2}$, respectively. The value of α corresponds to $C_0 \approx 3 \times 10^{-14}$ F, which in turn gives an estimate value of $\beta \approx 0.075 \text{ (mV)}^{-1/2}$. The agreement of the predicted functional relation between V_0 and V_c with that of the data in Fig. 4(b) is very encouraging, although the agreement may have been fortuitous in such relatively narrow ranges of V_0 and V_c . Taking the contribution from quasiparticle tunnelings into account does not change the linear functional relation between $\sqrt{V_0}$ and V_c , although it will change the values of the coefficients α and β . There is no way to determine C_0 quantitatively. We estimate the self-capacitance from the relation $C_0 = 4\pi\epsilon_0\kappa a$ to be about 4×10^{-16} F, where $\kappa = \epsilon[1 + a/(2s)] = 135$ with the permittivity of indium oxide¹³ of $\epsilon \approx 10$, $a \approx 25$ nm, and the inter-grain spacing of $s \approx 1$ nm. We see that there is much discrepancy between the estimated value of C_0 and the observed one. Quantitative comparison to the observed values in this case does not mean much, because of the crudeness of the analysis. Nonetheless, the fair agreement between data and the predicted relation seems to indicate that the insulating phase indeed originates mainly from the Coulomb blockade of pair tunnelings between superconducting grains. The reduced rate of pair tunneling due to the Coulomb blockade in turn induces fluctuations in the intergrain phase coupling, consistent with the picture of the scaling theory.

In Fig. 5 we plot the values of V_c as a function of magnetic field for temperatures of 0.403 K and 0.096 K. For a given magnetic field the values of V_c are insensitive to the temperature. In a field smaller than 7.2 T the magnetic field dependence of V_c becomes almost saturated but they decrease rapidly with field above 7.5 T and presumably vanish around 7.8 T. In Fig. 5 we also illustrate the boundary of the conducting-to-insulating transition due to grain charging in the temperature-field (T - H) diagram, where the data points

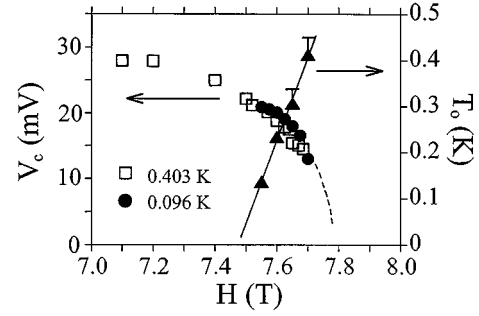


FIG. 5. Phase boundaries defined by the field dependences of V_c and T_0 , where T_0 is the onset temperature of the zero-bias peak in the differential resistance due to Coulomb blockade in a given magnetic field. The straight is a best fit line to the T_0 - H data.

were determined from the critical temperature for the appearance of the zero-bias Coulomb peak in a given magnetic field. The values of the critical magnetic field on the boundary decrease almost linearly with decreasing temperatures, implying that at lower temperatures the insulating state will develop in a smaller magnetic field than at higher temperatures. By extrapolating the phase boundary we estimate the value of the critical field H_c to be about 7.47 T at zero temperature. The value of this presumable zero-temperature critical magnetic field of the SI transition is definitely smaller than the one determined from the R vs T data in Fig. 1 or Fig. 2. Thus, we find in the figure that the local superconductivity exists in the grains for the critical field of SI transition at the lowest temperature used in the experiment (0.096 K) as well as in the zero-temperature limit, which is in accordance with the assumption of the scaling theory.³ But in the insulating state in magnetic fields above $H > 7.8$ T, no local superconductivity exists inside the grains, the state of which may correspond to the Fermi-glass phase observed in more uniformly disordered indium oxide composite films.⁶ In this sense, the insulating state in $H > 7.8$ T is totally different from that studied previously in more disordered insulating granular indium oxide films, where the local superconductivity inside grains persists even deep in the insulating regime.¹⁹

IV. SUMMARY

In summary, we carefully examined the magnetic-field-induced SI transition in In/InO_x granular films. The resistive transition reveals a quasireentrant behavior in the field range around 7.5 T, which are typical characteristics of granular systems. In a higher field around 8–9 T at low temperatures the resistance of the films exhibits typical temperature dependence of weak localization. Corresponding to the quasireentrant region of temperature and field, the DR shows a crossover from a superconducting Josephson-gap behavior to an insulating charging-gap behavior. The charging-gap behavior presumably originates from the Coulomb blockade of Cooper pairs between grains where the local superconducting order remains finite. In our samples, which can be considered as typical granular systems, the enhanced phase fluctuations due to Coulomb blockade of Cooper pairs as exhibited by the zero-bias DR peak destroy the global phase coherence between superconducting grains and lead to SI

transition. Due to the thermal effect the field-driven SI transition at a lower temperature takes place at a lower magnetic field. This study clearly reveals that in the granular films the phase fluctuations play a major role right at the SI transition, as predicted by the scaling theory. However, since the amplitude of the order parameter is highly suppressed by a high magnetic field near the SI transition the effect of amplitude fluctuations of the order parameter should be fully taken into account in order to understand the characteristics of the field-driven SI transition in granular systems.

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